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Effect of wall thermal conductivity on the heterogeneous combustion of hydrogen-air mixtures over platinum in catalytic micro-combustors

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ABSTRACT

The hetero-/homogeneous combustion of hydrogen-air mixtures over platinum in catalytic micro-combustors were investigated numerically and experimentally. Experiments were performed in a catalytic channel-flow micro-reactor. Catalysts consisting of platinum supported on alumina powder were prepared and tested. Numerical simulations were carried out with a two-dimensional computational fluid dynamics model in conjunction with detailed heterogeneous and homogeneous reaction mechanisms. Specific objectives are to study the effects of combustor wall conductivity on heterogeneous combustion characteristics and the steady-state, self-sustained flame stability of hydrogen-air mixtures. Hydrogen-air heterogeneous flame is found that it can be stabilized in narrow channels but very careful design is necessary. Large axial and transverse gradients are observed even at these small scales under certain conditions. Wall thermal conductivity is vital in determining the flame stability of the system, as the walls are responsible for the majority of the upstream heat transfer as well as the external heat losses. Hydrogen heterogeneous flames are more robust than methane heterogeneous flames. The catalytic self-ignition nature of hydrogen-air mixtures offers an opportunity to catalytic self-ignite hydrocarbons, resulting in a way toward elimination of ignition sources from micro-scale devices. Design recommendations are made, engineering maps denoting flame stability are constructed, and comparisons with methane-air systems are made.

Keyword: Micro-combustion; Heterogeneous combustion; Flame stability; Micro-combustion; Supported platinum catalysts; Wall thermal conductivity; Computational fluid dynamics

INTRODUCTION

Recently, there has been an increased interest towards the production of micro-scale power devices for portable applications, with efforts aiming at providing an alternative to the relatively low power density standard Li-ion batteries [1]. A common approach in many such attempts is the use of hydrocarbon fuels, because of their high energy content that renders even a relatively inefficient device capable of delivering high energy densities [2]. An option for utilizing the chemical energy of hydrocarbons is via a portable micro-compressor, micro-turbine, and micro-generator arrangement [3]. As the scale of the device is reduced, however, the resulting increase in surface-to-volume ratio gives rise to increased heat losses and radical quenching on the reactor walls. Heterogeneous (catalytic) combustion is a plausible solution to overcome such issues, due to it satisfies the requirements of large surface-to-volume ratios and moderate operational temperatures relevant to micro-scale devices [4].

Heterogeneous combustion methodologies are of interest in power generation due to their enhanced combustion stability at very lean equivalence ratios and the ensuing ultra-low nitrogen oxide emissions [5]. In the conventional CST (catalytically stabilized thermal combustion) mode [6], fractional fuel conversion is achieved in a heterogeneous (catalytic) reactor, which is typically palladium or platinum-coated and is operated at fuel-lean stoichiometry, while the remaining fuel is combusted in a follow-up homogeneous (gas-phase) combustion region, again at fuel-lean stoichiometry [7]. Applications of heterogeneous combustion at the micro-scale also include stationary gas turbines of large power plants, whereby combustion is attained in honeycomb reactors comprising a multitude of catalytically coated channels [8]. Heterogeneous combustion methodologies crucially depend on advances in catalyst technology (development of active and thermally stable catalysts) as well as on multidimensional modeling needed for reactor design, with the latter being the focus of the present work.

When miniaturizing catalytic combustors from the macro-scale down to the micro-scale, some phenomena start playing a relevant role, such as heat losses toward the external environment and transverse diffusion with respect to the residence time. These phenomena may significantly affect the thermal behavior of catalytic micro-reactors. Benedetto *et al.* [9] performed three-dimensional computational fluid dynamics simulations for studying the effect of the cross-sectional geometry on the ignition/extinction behavior of catalytic micro-combustors. Two different shapes of the cross-section were investigated, square and circular. Stability maps were built and compared for lean propane-air combustion at different inlet gas velocities. They found that, at low inlet velocities, the square cross-section channel is more resistant to extinction than the cylindrical channel. Close to the ignition point, a strong coupling between fluid flow and superficial reaction rate is established. For both geometries, this coupling does not allow blowout to occur when increasing the inlet velocity up to the value limiting the laminar regime for the incoming flow. Merotto *et al.* [10] presented a novel catalytic micro-scale combustor fueled with propane-air mixture, coupled with two conventional thermoelectric modules. The wafer-like

combustor is filled with commercially available catalytic pellets of alumina with platinum. Temperature measurements, in terms of point values and two-dimensional distribution across the combustor surfaces, were carried out in different operating conditions. Exhaust gases concentration measurements and pellet aging investigation were performed to determine the combustor efficiency and stability. Starting from these results the combustor has been coupled to bismuth telluride-based thermoelectric modules using a water cooled heat exchanger at the cold side. The system obtained produces 9.86 W of electrical power reaching an overall efficiency up to 2.8%: these results represent an improvement in portable-scale electrical power production from hydrocarbon fuels state-of-art. Moreover, the voltage and current characteristics allow using such generator for small portable devices power supplying.

Hsueh *et al*. [11] performed a numerical study to examine the characteristics of heat and mass transfer and the performance of a plate methanol steam micro reformer with a methanol catalytic combustor. In addition, the effects of the flow configurations for co- and counter-current flows were explored. The influences of the Reynolds number and various geometric parameters on heat and mass transfer phenomena in the channels were also investigated numerically. It is shown that the Reynolds number and various geometric parameters can be improved by thermal management to enhance the chemical reaction and thus augment the micro reformer performance. Comparing the co- and counter-current flows via numerical simulation, the results show that the methanol conversion for counter-current flow could be improved slightly. This is because of the fact that counter-current flow results in a better thermal management, which in turn improves fuel conversion efficiency. With a higher Reynolds number on the combustor side, the wall temperature is increased and the methanol conversion can consequently be enhanced. Meanwhile, a reduced Reynolds number on the micro reformer side would increase the methanol conversion. The results also reveal that appropriate geometric parameters exist for a micro reformer with a combustor to obtain better thermal management and methanol conversion. Wierzbicki *et al*. [12] examined the oxidation behavior of dodecane and two mixtures of dodecane and m-xylene over an Rh catalyst in a meso-scale heat recirculating combustor to isolate the effect of aromatic content on performance. The fuel conversion, product selectivities, and reaction kinetics were calculated, and the global combustion behavior observed. The results showed that increasing the amount of m-xylene in the fuel increased the fuel conversion from 85% to 92% and further to 98%. The presence of xylene also significantly increased CO_2/H_2O selectivity and decreased CO/H_2 selectivity. Global activation energy increased linearly with increase in xylene content, supporting that addition of aromatic species to fuel lowers the overall reactivity. The non-catalytic reaction was also simulated using CHEMKIN software to determine the effect of the Rh catalyst on the combustor performance and to analyze the difference in chemical mechanisms. The results revealed that the catalyst promotes total oxidation over partial oxidation, and lowers the global activation energy by up to 70%.

Despite previous work, there are a number of answered questions regarding the hetero-/homogeneous combustion of hydrogen-air mixtures over noble metals at the micro-scale. In this work, the hetero-/homogeneous combustion of hydrogen-air mixtures over platinum in catalytic micro-combustors were investigated numerically and experimentally. Experiments were performed in a catalytic channel-flow micro-reactor. Numerical simulations were carried out with a two-dimensional elliptic computational fluid dynamics model in conjunction with detailed multicomponent transport, detailed heterogeneous and homogeneous reaction mechanisms, and heat transfer mechanisms.

Model

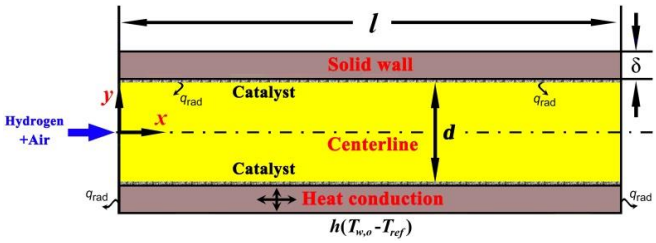

Fig. 1. Schematic diagram of the channel-flow catalytic micro-combustor geometry.

The channel-flow catalytic micro-combustor consists of two infinite parallel plates coated with platinum catalyst, 4.0 mm long, separated by a gap distance *d*, as shown in Figure 1. Due to the aspect ratio, the combustor is modeled as a two-dimensional system, which involves writing conservation equations in the axial direction and a lumped parameter description of transverse heat and mass transfer. The parallel plate geometry implies that the third dimension (width) of the micro-combustor is much larger than the gap size; wherever relevant. Hydrogen-air mixtures of varying concentrations are fed into the reactor at ambition temperature. The following assumptions are made: the flow is laminar, the pressure drop is negligible, gases follow the ideal gas law, and radiation effects are neglected due to the large aspect ratio of the reactor. Symmetry was not invoked in order to allow for potential asymmetric flame structures, as reported in previous non-catalytic channel combustion studies [13].

The governing equations were solved for a steady laminar reactive flow in two-dimensional homogeneous micro-combustors. Simulations were performed by using the computational fluid dynamics software, FLUENT® Release 6.3 [14] was used to perform these simulations. A finite-difference method is used to discretize the two-dimensional continuity, momentum, energy and species conservation equations in the fluid and the two-dimensional energy equation in the wall.

The oxidation of hydrogen on platinum was described by the detailed surface reaction scheme of Deutschmann *et al*. [15] with a surface site density of $\Gamma = 2.7\times10^{-9}$ mol/cm^2, consisting of 14 elementary reactions, 6 gaseous and 5 surface species. The gas-phase chemistry was described by the detailed mechanism of Li *et al*. [16] containing 9 gaseous species participating in 21 reversible elementary reactions. The capacity of this scheme to reproduce measured gas-phase ignition characteristics under both fuel-lean and fuel-rich hydrogen-air hetero-/homogeneous combustion has been demonstrated in the work of Ghermay *et al*. [17].

Gas-phase and surface reaction rates were evaluated with CHEMKIN [18] and Surface-CHEMKIN [19], respectively. Mixture-average diffusion, including thermal diffusion, was used in conjunction with the CHEMKIN transport database [20].

Dirichlet boundary conditions are imposed at the reactor entrance, and zero-flux boundary conditions are applied at the reactor outlet. At the interface between the solid and the fluid, no slip and no normal species diffusive flux boundary conditions are employed.

The heat flux at this interface is calculated using Fourier's law and continuity in temperature and heat flux is ensured. In the bulk of the wall the two-dimensional energy equation is solved. The exterior/top surface of the wall is assumed to obey Newton's law of cooling. It is important to note that all the two-dimensional internal heat transfer within the fluid and the solid are calculated explicitly with the two-dimensional elliptic models without any further simplifications. The exterior convective heat-transfer coefficient is only used for the calculation of the heat flux of the exterior wall edge boundary condition. This heat-transfer coefficient lumps the details of heat loss from the micro-combustor and of the process that utilizes the heat generated by the combustor.

The simulations were performed on a Beowulf cluster consisting of 6 × Xeon X5670 processors and 80 GB of RAM. When parallel processing was used, the message passing interface (MPI) was used to transmit information between nodes. In order to achieve convergence as well as compute extinction points, natural parameter continuation was implemented. The calculation time of each simulation varied between 8 hours and several days, depending on the difficulty of the problem and the initial guess.

EXPERIMENT

Supported platinum catalysts were prepared by the incipient wetness impregnation technique using $Pt(NO_3)_2$ as metal precursor [21]. The supports had previously been calcined at 980 K for 8 hours. The only exception was alumina, which was calcined at 1480 K for 8 hours to obtain the α-Al_2O_3 phase. The impregnated powders (0.5-2 wt% noble metal loading) were dried at 380 K and calcined at 880 K for 6 hours before coating on FeCr-alloy metal. The coated FeCr-alloy was assembled to a fully coated micro-channel structure with a diameter of 0.8 mm a total length of 4 mm, containing about 0.8 g of catalysts.

The experiments were carried out in a micro-channel test rig. Sheathed thermocouples monitored the temperature at the reactor entry and exit and at different axial locations of dedicated catalytic channels. The catalyst was not externally heated or cooled and, consequently, the reactor operated under near adiabatic conditions. All reactants and products except water were analyzed by gas chromatography. The water concentration was calculated using element balances. A gas mixture was fed to the reactor, which operated at ambition pressure and a fixed GHSV (gas hourly space velocity). The flow rate of hydrogen was varied in order to obtain different oxygen-to-fuel ratios.

The BET (Brunner-Emmett-Teller method) surface area of the catalyst powders was measured by nitrogen adsorption using a Micromeritics ASAP 2020 - Physisorption Analyzer. The samples were degassed at 580 K prior to analysis. TPR (temperature programmed reduction) experiments were performed on a Micromeritics AutoChem II 2920 Chemisorption Analyzer. The samples were pretreated in argon at 480 K for 38 minutes before reduction. The reduction was carried out in 8% hydrogen-argon by heating the sample from room temperature to 980 K at a heating rate of 8 K/min. The noble metal dispersion was determined by hydrogen chemisorption analysis performed on a Quantachrome Autosorb-iQ-TPX. The samples were reduced in hydrogen at 680 K for 48 minutes followed by evacuation prior to the analysis. The adsorption measurements were performed using hydrogen as adsorptive. The lower adsorption temperature was used in order to suppress spill-over of hydrogen to the support, which has been reported to occur for ceria-containing supports under certain conditions [22]. The metal dispersion was calculated according to the dual isotherm method.

Catalyst-coated FeCr-alloy samples were analyzed by XPS (X-ray photoelectron spectroscopy) and Raman microscopy. Selected samples were analyzed both before and after the activity tests to determine any structural changes occurring under reaction conditions. XPS experiments were performed in a VG Thermo Escalab 220i-XL X-ray photoelectron spectroscopy system, using Mg Kα radiation. The electron analyzer was used in the constant pass energy mode with a pass energy of 20 eV. The pressure in the analysis chamber during the measurements was always better than 8 mbar. The composition of the surface and sub-surface regions accessible by XPS was calculated from the intensity of the XPS peaks using the transmission function of the electron analyzer. Charging during the XPS experiments was in all cases smaller than 8 eV. Charge correction was carried out setting the peak of adventitious carbon to a value of 248.0 eV.

RESULTS AND DISCUSSION

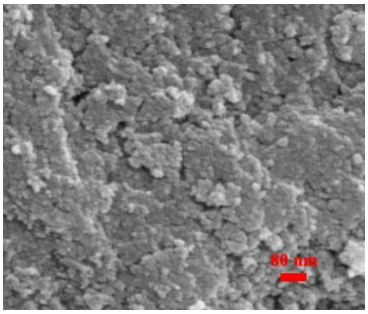

Fig. 2. SEM image of the alumina powder supported platinum catalyst samples.

Several typical samples have been chosen for scanning electron microscopy (SEM) analysis to study the effect of the impregnation and reduction processes on the catalyst properties. Figure 2 shows the surface morphology of alumina powder supported platinum catalyst. The particle size of the alumina is about 60 nm, and platinum particles are too small to be distinguished.

Norton and Vlachos introduced the flame location as a convenient criterion for the stability or robustness of a micro-combustor, defined as the axial position with the highest reaction rate [23]. Raimondeau *et al*. [24] presented that, in very small reactors, radial gradients and temperature discontinuity at the wall are negligible but become significant as the diameter is increased. On the other hand, the near-entrance heat loss and radical quenching at the wall are crucial issues in controlling flame propagation in micro-channels.

Thermal conductivity is the thermal property of the combustor wall to conduct heat, and is evaluated primarily in terms of Fourier's Law for heat conduction. Heat transfer occurs at a lower rate across the combustor wall of low thermal conductivity than across the combustor wall of high thermal conductivity. Correspondingly, the combustor wall of high thermal conductivity is widely used in heat sink applications and the combustor wall of low thermal conductivity is used as thermal insulation. The thermal conductivity of combustor wall may depend on temperature. The reciprocal of thermal conductivity is called thermal resistivity. Thermal conductivity is actually a tensor, which means it is possible to have different values in different directions.

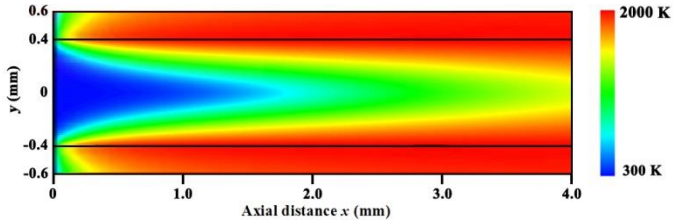

Fig. 3. Two-dimensional temperature distributions for low wall thermal conductivity.

Figure 3 shows contour plots of the temperature for low wall thermal conductivity $k = 0.8$ W/m^2 K. The entire micro-combustor is shown. The flame stabilizes in the center between the two plates. The heterogeneous reaction starts at the catalytic wall and travels towards the center as the flow goes downstream. Heterogeneous combustion occurs very rapidly, consuming most of the hydrogen in a very small region. Complete conversion is achieved, and a significant temperature rise is observed due to the exothermicity of the heterogeneous reaction. For low wall thermal conductivity, the flame location is considerably downstream and blowout may occur depending on burner length and flow velocity. For cases of low wall thermal conductivities, the upstream heat flux through the wall is limited, causing low upstream wall temperatures. This in turn results in slower preheating of the fluid to the ignition temperature, which shifts the flame location downstream. Lack of upstream heating is one mechanism of loss of flame stability. The narrow flame front observed in the simulations is consistent with the experimental observations by Churchill of a large-diameter thermally stabilized combustion (TSB) [25]. They found that thermally stabilized combustion has a number of unique characteristics which permit the generation of steam or other forms of process energy from the heat of combustion of a gaseous or clean liquid fuel in remarkably compact, integrated apparatus while truly minimizing the concentrations of nitrogen oxide, carbon monoxide and unburnt fuel in the effluent; the wall temperature profile is the primary determinant of the stability of the flame in micro-channels.

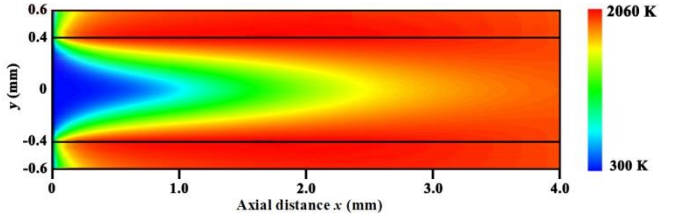

Fig. 4. Two-dimensional temperature distributions for moderate wall thermal conductivity.

Figure 4 shows contour plots of the temperature for moderate wall thermal conductivity $k = 8$ W/m^2 K. As the wall thermal conductivity increases to high values, the flame location shifts upstream. As the thermal conductivity is slightly increased to moderate wall thermal conductivity, typical of ceramics, a drastic reduction in flame location from the entrance is observed. Simulation results indicate that some moderate wall conductivity is essential for transferring heat upstream to cause ignition and stabilize the flame near the micro-combustor entrance. Therefore, the location of the flame shifts as a function of operating parameters. The results are consistent with the simulation observations by Norton and Vlachos [26], who used a two-dimensional elliptic computational fluid dynamics model of a non-catalytic micro-combustor to examine the effects of combustor dimensions, external heat losses, wall thermal conductivity, and operating conditions on combustion characteristics and the steady-state, self-sustained flame stability of hydrogen-air mixtures. The results illustrated that the wall thermal conductivity is vital in determining the flame stability of the system, as the walls are responsible for the majority of the upstream heat transfer as well as the external heat losses. In addition, there exists a range of flow velocities that allow stabilized combustion in non-catalytic micro-combustors. Furthermore, the combustor dimensions strongly affect thermal stability.

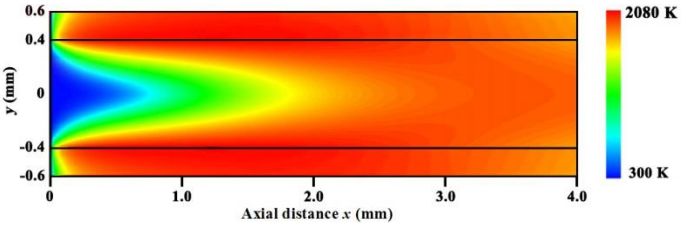

Fig. 5. Two-dimensional temperature distributions for high wall thermal conductivity.

Figure 5 shows contour plots of the temperature for high wall thermal conductivity $k = 80$ W/m^2 K. Despite the small scales of these systems, large transverse gradients in temperature exist in the fluid and large axial gradients in temperature may exist in the walls. For high wall thermal conductivity and low external-heat-loss coefficients, increasing wall thermal conductivity to high values has a minor effect on the flame location (not shown). However, for high external-heat-loss coefficients of the catalytic micro-combustor, increasing wall thermal conductivity shifts the heterogeneous reaction downstream. This nonlinear behavior is caused by the interaction between two competing modes of heat transfer, i.e., upstream heat transfer through the walls to preheat the feed, and transverse heat transfer resulting in heat loss to the surroundings. The former is critical for ignition and flame stabilization in catalytic micro-combustors, as it allows preheating of the feed without the need for an external preheater. If the upstream heat transfer is insufficient to increase the fluid temperature to the ignition temperature, a flame is not stabilized within catalytic micro-combustors [27]. Since the thermal conductivity of the walls is orders of magnitude higher than that of the fluid, heat conduction through the walls is the primary mechanism of upstream heat transfer. When this upstream heat transfer is limited by low wall thermal conductivity, it takes a greater distance to achieve the preheating, resulting in the reaction region shifting downstream. This makes the flame less stable. For a given wall thermal conductivity, increasing the external heat loss coefficient shifts the reaction region downstream as more of the heat generated is lost to the surroundings.

Parametric continuation is used to move from one stationary solution to another. When the solution reaches a turning point or blowout occurs, this is denoted as a critical point. Knowledge of critical parameter values of the external heat transfer coefficient, wall thermal conductivity, feed composition, and flow velocity gives a better understanding of the important factors controlling flame stability. These critical values are useful as guides, but

actual values will vary depending on the system (e.g., dimensions) of interest [28].

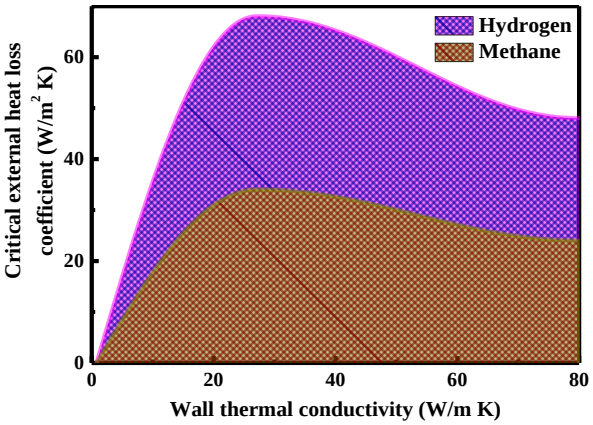

Fig. 6. Critical external heat loss coefficient as a function of wall thermal conductivity. Typical ceramics allow maximum external heat loss coefficients. Combustor walls with lower thermal conductivities limit the upstream heat transfer. Combustor walls with higher thermal conductivities result in enhanced heat transfer to the surroundings. Hydrogen allows self-sustained heterogeneous combustion for higher external heat loss coefficients and more insulating combustor walls than methane.

Figure 6 shows the critical external heat loss coefficient as a function of wall thermal conductivity. These bell-shaped envelopes separate the region of self-sustained heterogeneous combustion below the curve from the region above the curve where heterogeneous combustion cannot be self-sustained. There exists a critical wall thermal conductivity for hydrogen-air mixtures, at approximately 0.8 W/m K, below which heterogeneous combustion cannot be self-sustained, even with insulating walls. When the wall thermal conductivity increases from low values, the allowable-heat-loss coefficient first increases quickly, and then decreases and levels off in the range of metals or high-thermal-conductivity ceramics such as silicon carbide. The allowable-heat-loss coefficient reaches a maximum for insulating ceramics such as silica and alumina. The behavior seen for low-conductivity combustor walls is at first counterintuitive. Highly insulating combustor walls are poor for flame stability due to the lack of a continuous heterogeneous ignition source, needed to preheat the cold incoming gases.

The mechanism of hydrogen for the loss of stabilized heterogeneous combustion is qualitatively similar to that of methane. For low wall thermal conductivities the primary mode of burner instability is blowout, whereas for high wall thermal conductivities it is extinction. However, hydrogen heterogeneous flames are more robust than methane heterogeneous flames. Note that the methane map is a subset of the propane map. Lower wall thermal conductivities and higher exterior heat-loss coefficients are possible. In fact, hydrogen-air mixtures are known to be catalytic self-igniting over platinum foils and wires under very fuel-lean conditions, but fuel-richer mixtures exhibit ignition temperature above room temperature. The catalytic self-ignition nature of hydrogen-air mixtures at the small scale of ceramic burners offers an opportunity to catalytic self-ignite hydrocarbons. This concept may be a way toward elimination of ignition sources from micro-scale devices, resulting in further reduction of system dimension. Furthermore, since hydrogen is a main target for fuel cell applications, one can envision storage of small amounts of hydrogen during device operation from reforming of hydrocarbons that is subsequently used for startup.

CONCLUSIONS

The hetero-/homogeneous combustion of hydrogen-air mixtures over platinum in catalytic micro-combustors were investigated numerically and experimentally. Experiments were performed in a catalytic channel-flow micro-reactor. Numerical simulations were carried out with a two-dimensional elliptic computational fluid dynamics model in conjunction with detailed multicomponent transport, detailed heterogeneous and homogeneous reaction mechanisms, and heat transfer mechanisms. We have found that hydrogen-air flames can be stabilized in narrow channels but very careful design is necessary. The combustor wall thermal conductivity plays a competing role in flame stability. Combustor walls transfer heat upstream for ignition of the cold incoming gases but at the same time are responsible for heat losses. Despite the small scales of these systems, large transverse gradients in temperature exist in the fluid and large axial gradients in temperature may exist in the walls. In addition, hydrogen heterogeneous flames are more robust than methane heterogeneous flames. They allow a wider range of wall thermal conductivities as well as higher external-heat-loss coefficients. The catalytic self-ignition nature of hydrogen-air mixtures at the small scale of ceramic burners offers an opportunity to catalytic self-ignite hydrocarbons. This concept may be a way toward elimination of ignition sources from micro-scale devices, resulting in further reduction of system dimension. Furthermore, since hydrogen is a main target for fuel cell applications, one can envision storage of small amounts of hydrogen during device operation from reforming of hydrocarbons that is subsequently used for startup.

Acknowledgments

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